US EPA RECORDS CENTER REGION 5

August 4, 1982

Michael J. Hansel Regulatory Compliance Section Solid and Hazardous Waste Division Minnesota Pollution Control Agency 1935 West County Road B2 Roseville, Minnesota 55113

Dear Mr. Hansel:

The first revision of the QA Project Plan for the PAH measurements is enclosed. The items that were revised included reducing the number of target compounds, identifying the states authorized agent, identifying the specific internal standards and surrogate standards to be used, and limiting item 15.1.1 to just the PAH internal standards.

When CH<sub>2</sub>M Hill completes another iteration of the Method Detection Limit. Table 3 will be revised. Then only the final revision of Table 3 and the Table of Contents need be included in the QA Project Plan to complete the document.

I can be reached at 513-684-7311.

Sincerely yours,

Denis L. Foerst, Research Chemist Organic Analyses Section Physical and Chemical Methods Branch

Enclosure: As stated.

cc: Harold Cole, w/enclosure
Michael Kosakowski, w/enclosure
Paul Better, w/enclosure
Brenda Kimble, w/enclosure
Eugene Meier, w/enclosure

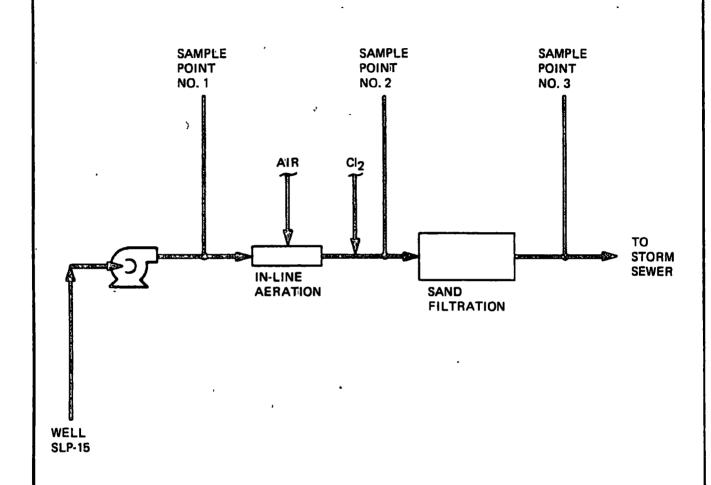


FIGURE E-1 FLOW SCHEMATIC EXISTING TREATMENT SYSTEM AT WELL SLP-15



L16334.BO.01

# CHRONOLOGICAL RECORD OF WELL WATER ANALYSES

	AMETER							-	
ALL	VALUES IN NG/L)	9/15/32	9/15/82	9/15/82	9/15/82	9/15/82			
		WELL HEAD	AFTER AIR	AFTER CL2	2 DAYS AFTER	5 DAYS AFTER FILTRATION	•	į	
ANA	LYSIS BY:	CH2M	CH2M HILL	CH2M	CHZM	CH2M HILL			
	ANALYTICAL EQUIPMENT	HILL	GCMS	HILL	HILL	GCMS	<del></del>	<del> </del> -	
	ACENAPHTHENE	2200	1000	880	520	400		ļ	_
Ì	ACENAPHTHYLENE	1200	550	450	240	200	<del></del>		_
ł	ANTHRACENE	130	21	720	270	200	<del>_</del>		
1	BENZO(a)ANTHRACENE *	120	<u> </u>		<del>                                     </del>	-		<del> </del>	
1	BENZO(b&k)FLUORANTHENE*		<del></del>			<del> </del>		<del></del>	
⊋ Ì	BENZO(g, h, ı)PERYLENE			<del> </del>	<del> </del>	l ~		<del></del>	
₹	BENZO(a)PYRENE®	<del></del>		<del> </del>		<del>   </del>			
၌ {	BENZO(e)PYRENE			<del>                                     </del>	<del> </del>			<del> </del>	
z I	CHRYSENE *		<del> </del>	<del> </del>	<del> </del>	<del> </del>		<del> </del>	_
<u> </u>	DIBENZO(a, h)ANTHRACENE*		<del> </del>		<del> </del>	<del> </del>		<del>                                     </del>	-
2	FLUORANTHENE	430	21	<del> </del>	6.2			<del> </del>	
(*INDICATES CARCENOGENIC PAH)	FLUORENE	2100	670	570	210	170		<del> </del>	
A B	INDENO(1, 2, 3-cd)PYRENE*	2100	1010	1 2/0	- 210	<u>''</u>		<del> </del>	
ပ္က	I-METHYLNAPHTHALENE	93	. 63	58	28	23		<del>                                     </del>	
ű	2-METHYLNAPHTHALENE	/2	17	1 70	63	<del>  </del>		<del></del>	-
፳	NAPHTHALENE	17	940	740	94	86			
ᇊ	PERYLENE		· / <del>-</del> -0	1-1-0	<del>- /</del>	20			
Ξ	PHENANTHRENE	100	31	<del> </del>	5.3				-
	PYRENE	340	16	<del></del>	5.2	<del> </del>	<del></del>	<del>                                     </del>	
PAH	1, 2, 6, 7-TETRAHYDROPYRENE	2-10	1	<del></del>		f		<del></del>	
4	9. 10-BENZPHENANTHRENE		,						
	<b></b>				<b> </b>	<b> </b>		<b> </b>	_
					ļ. <u></u>	<b></b>			
	TOTAL CARGENOGEN		ļ	<del> </del>	<del> </del>	<b> </b>	<del></del>	<u> </u>	
	TOTAL CARCENOGENIC PAH'S		- 0200	-		- 070		<b>}</b>	
	TOTAL "OTHER" PAH'S	6610	3329	2698	1172	879		<u> </u>	_
ES	ACRIDINE			<del> </del>	ļ	<del>  </del>		<del> </del>	
교리	CARBAZOLE			<del> </del>		<del> </del>			-
الح ق	INDOLE	<u> </u>		ļ	ļ	<del> </del>		<del> </del> -	_
200	PHENANTHRIDINE	ļ	20	7/	<del>                                     </del>	<del>  ,,  </del>		<u> </u>	
NITROGEN HETEROCYCLES	QUINOLINE		32	76	11	11		<u> </u>	
<b>-</b> #		<del> </del>	<del> </del> -	<del> </del>	<del> </del>	<del>  </del>		<del>├</del> -	
<del></del>	BENZO(b)THIOPHENE	390	320	320	120	110		<del> </del>	
S HET.	BENZO(0)THIOPHENE	<u> 570</u>	320	320	120	110		ļ	
Ï		<del> </del>	<del> </del>		<del> </del>	<del>  </del>		<del> </del>	
	BIPHENYL	540	260	290	70	59		<del> </del>	
US	2, 3-DIHYDROINDENE	1700	1600	1600	890			<del> </del>	-
MISCELLANEOUS	INDENE	72	50	50	29	25		<del> </del>	-
Z		1.5	1	<del> </del>		<del>  </del>		<del> </del>	
3		<del>                                     </del>	<b></b>		<del> </del>	<del>  </del>		<del> </del>	
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AROMATIC AMINES		<u> </u>		<b> </b>					
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S S			T	T					
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Quality Assurance Project Plan for the Measurement of PAH Compounds at ng/L Levels by Gas Chromatography/Mass Spectrometry

Prepared for CH2M Hill

Denis L. Foerst, Research Chemist Organic Analyses Section Physical and Chemical Methods Branch Robert L. Booth, Acting QA Officer Environmental Monitoring and Support Laboratory - Cincinnati

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## 3. PROJECT DESCRIPTION

CH2M Hill is to conduct a project to measure the PAH concentration at ng/L levels in:

- the ground water in the vicinity of Saint Louis Park, Minnesota,
- the influent, the effluent, and various stages of the existing treatment facility,
- various stages during a series of bench scale treatments,
- the influent and effluent of a pilot plant during a 30 day study.

The analytical procedure involves the serial extraction of the aqueous sample with methylene chloride at pH >11 and then pH <2, concentration, and analysis via capillary column gas chromatography/mass spectrometry (GC/MS).

The anticipated sampling schedule is given in Table 1. The target compounds are listed in Table 2. The PAH measurement data will be used to judge the treatability of the selected treatment process.

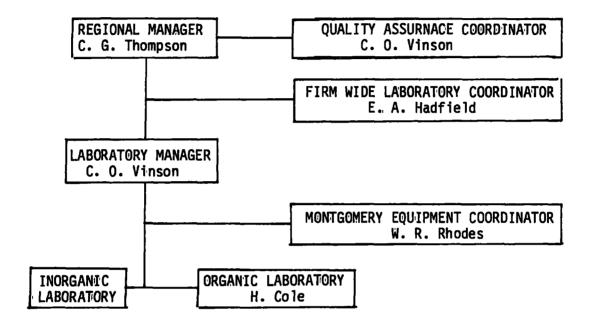
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#### 4. PROJECT ORGANIZATION AND RESPONSIBILITY



- 4.1 The Regional Manager will review all QA data with the Laboratory Manager on a quarterly basis.
- 4.2 The Laboratory Manager is responsible for the continuity and control of the QA program.
- 4.3 The Quality Assurance Coordinator is responsible for:
  - 4.3.1 Logging samples and introducing control samples.
  - 4.3.2 Monitoring QA activities.
  - 4.3.3 Informing the staff and management of non conformance to the QA program.
  - 4.3.4 Reviews purchased materials to ensure that quality materials are purchased.
  - 4.3.5 Receives data prior to reporting and maintains QA documents.

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# 5. QA OBJECTIVES FOR PAH MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS AND METHOD DETECTION LIMITS

- 5.1 The QA objective for precision is an average relative range for duplicate analyses of less than 30% at a 95% confidence level. The preliminary validation study indicates that the relative standard deviation of laboratory control standards exhibits a slight concentration dependence (Figure 1).
- 5.2 The QA objective for accuracy is an average bias for the spiked samples of less than 25%. The preliminary validation study exhibited an average bias of -8% and -18% for 15 PAH compounds for true values of 10 ng/L and 50 ng/L respectively.
- 5.3 The QA objective for completeness is 90%. No more than 10% of the data is to be ruled invalid due to QA/QC checks on the overall system performance.
- 5.4 The QA objective for method detection limit (MDL) is an average MDL of less than 5 ng/L. The validation study gave an average MDL, for 15 PNAs, of 4.7 ng/L. (Table 3)

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## 6. SAMPLING PROCEDURE

- 6.1 Method 624, purgeables, requires a duplicate sample to be collected and preserved with acid if analysis is to be performed between 7 and 14 days after collection due to the potential biological degradation of benzene, toluene, and ethylbenzene. If not acid preserved, the purgeable samples must be analyzed within 7 days.
- 6.2 The PNA compounds are susceptible to photodegradation, therefore, amber containers or foil wrapped containers must be used. Extraction must be completed within 7 days of collection. Extracts must be analyzed within 40 days of extraction.
- 6.3 Sample containers must be scrupulously cleaned. All sample containers are to be washed with detergent, rinsed with tap water, reagent water, and set aside to dry. PNA sample containers, after drying, are rinsed with a polar and a non-polar organic solvent and again set aside to dry before use.
- 6.4 Triplicates, duplicates and field blanks are included in each set of samples as scheduled on Table 1. The triplicate is collected at a clean well or at a treatment effluent. The duplicate is collected at a dirty well or at a treatment influent. The field blank is sent from the lab to the field and back to the laboratory with the other samples.
- 6.5 The composition of the duplicates and triplicates must be homogenous. Collect these samples in as short a period of time as possible. Fill each bottle of a duplicate or a triplicate set by sequential thirds to ensure homogeneity.
- 6.6 When sampling inactive wells, record the number of well volumes that have been pumped prior to filling an individual sample. A minimum of 10 casing volumes should be pumped before collecting a sample.
- 6.7 When sampling an active well, record the number of gallons pumped in the previous 24 hours.
- 6.8 The specific sample tag is illustrated in Figure 2.
- 6.9 Field records must be completed at the time the samples are collected. The records must be signed or initialed including the date and time by each member of the sampling team. A Field Tracking Report Form is given in Figure 4.

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## 7. SAMPLE CUSTODY

- 7.1 Chain of custody procedures will apply to all samples. A chain of Custody Record form is given in Figure 5. All entries are to be completed in indelible ink. Dean Malotky is the field sampling team leader.
- 7.2 The original chain of custody record is sealed in a watertight plastic sandwich bag and shipped inside the sealed transportation case. A copy of the record is retained by the sampling team.
- 7.3 The samples are shipped to Harold Cole, the designated custodian at CH<sub>2</sub>M Hill. A permanent log book will be kept describing the samples as received. Log book entries are to include; the person delivering the sample, date and time received, source of sample, sample ID or log number, mode of transport, and the condition of the sample as received.
- 7.4 Samples are to be stored in the custody room, a securely locked area. Only the custodian is to deliver samples to the laboratory personnel. The laboratory is to be maintained as a secured area, restricted to authorized personnel only. Laboratory personnel are responsible for the care and custody of the sample after being received from the custodian. The sample must always be in the possession or view of the laboratory personnel or secured in the laboratory at all times until analysis is completed.
- 7.5 The unused portion of the sample, if any, and all identifying labels must be returned to the custodian. The custodian will retain unused portions of the sample until the State's Authorized Agent, Michael J. Hansel, authorizes that the unused samples are to be destroyed.

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## 8. CALIBRATION PROCEDURES AND FREQUENCY

- 8.1 The procedures for internal standard or external standard calibration are described in methods 624 and 625. The laboratory is responsible for demonstrating the linear range and the linearity of the calibration curve. If the concentration level of a target compound exceeds the linear range, the extract is diluted and reanalyzed for that compound.
- 8.2 The calibration of the GC/MS system is to be verified each day by 1) achieving the DFTPP or BFB key ion abundance criteria as appropriate, 2) achieving the benzidine or pentachlorophenol tailing factor criteria as appropriate, and 3) chromatographing an aliquot of the standard solution that contains the appropriate target compounds and updating the response factors if necessary.
- 8.3 Sources of the individual target compounds are given in Table 2. The source, purity, lot number, and certificate of true values for standard solutions will be recorded.

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## 9. ANALYTICAL PROCEDURES

- 9.1 Method 624 is to be used without change for the analysis of the purgeable samples.
- 9.2 The PNA compounds are analyzed using a procedure developed at CH<sub>2</sub>M Hill. This procedure is very similar to method 625 with the following exceptions:
  - 9.2.1 Two surrogate standards are used instead of three.
  - 9.2.2. The volume of the final extract is 0.02 mL instead of 1.00 mL.
  - 9.2.3 The internal standards are added just prior to the final concentration, subsequent analysis is performed immediately after this concentration. Method 625 calls for adding the internal standards just prior to analysis.
  - 9.2.4 The retention time agreement is to be  $\pm$  10 sec. instead of  $\pm$  30 sec.
  - 9.2.5 The MDL for the priority pollutant PNAs average less than 5 ng/L. Method 625 gives an average MDL of 3200 ng/L for the priority pollutant PNAs.

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## 10. DATA ANALYSIS, VALIDATION AND REPORTING

- 10.1 The area of each PNA internal standard (IS) is used to judge the validity of the assay step. The area of each PNA IS must be >20,000 counts. If the area is less than 20,000 counts, the GC/MS system must be retuned or the sample must be reanalyzed after additional concentration.
- 10.2 The recovery of the surrogate compounds is used to judge the validity of the sample processing steps. The surrogate standard recovery statistics are to be updated weekly to establish the control limits of R  $\pm$  3s. The sample processing steps are valid if the recovery for the surrogate compounds falls within the control limits.
- 10.3 The equations in Section 7 and 15 of Method 625 are to be used to calculate the concentration of the target compounds. Report "not detected" if the calculated concentration is less than the MDL. Report the MDL concentration if the calculated concentration is between the MDL and two times the MDL. Report the concentration in ug/L for purgeables or in ng/L for the PNAs if the calculated concentration is greater than two times the MDL.

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## 11. INTERNAL QUALITY CONTROL CHECKS

11.1 Field Blanks -- One field blank is included with each sample set.
Once received back in the laboratory, the field blank is treated and an authentic sample and is used to monitor for contamination during transport and sampling.

- 11.2 Laboratory Blanks -- A laboratory blank is analyzed whenever a field blank indicates the possibility of contamination or whenever a new lot of solvents is first used.
- 11.3 Surrogate Standards -- All samples, including blanks, are spiked with the surrogate standards prior to extraction and are used to monitor the sample processing steps. The surrogate standards are 1-fluoronaphthalene and 2,4,6-tribromophenol..
- 11.4 Internal Standards -- All extracts are spiked with the internal standards just prior to the final concentration. The internal standards are d-8 naphthalene, d-10 anthracene, d-12 chrysene, 2-fluorobiphenyl, and d-5 phenol.
- 11.5 Duplicates and Spiked Samples -- The duplicate pairs are used to give overall precision of the data in both a relatively clean and a contaminated matrix. The third sample of the triplicate is used to give spiked recovery or accuracy data. The background concentration is the mean value from the two unspiked samples of the triplicate. Since the spiked samples should always be relatively clean samples, a constant amount (100 ng) of each target compound should be used in all spiked samples.
- 11.6 Refereed Samples -- Samples sent out to the referee laboratories should include a field blank and a triplicate so that interlaboratory precision and accuracy can be compared. Capsule Labs will analyze samples using GC/MS, (modified Method 625), the Minnesota Department of Health will analyze samples using HPLC (modified Method 610), and EMSL-Cincinnati will analyze samples using HPLC method 610 and GC/MS method 625.
- 11.7 Quality Control Check Samples -- The analytical laboratories must compare calibration standards with the EPA QC check samples at least once during this study.

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# 12. PERFORMANCE AND SYSTEM AUDITS

12.1 Not applicable. No formal certification program or relevant interlaboratory performance evaluation study is available or planned for these compounds at the concentrations of interest. The data from the preliminary validation study will indirectly serve as the performance and system audits.

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# 1:3. PREVENTIVE MAINTENANCE

Not applicable — The system performance checks will show whether the participants' analytical systems are operable or not; the length of time necessary to do the required research does not warrant mandatory preventive maintenance programs. However, if any maintenance is performed – during the time frame of the project – then, that maintenance must be documented.

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# 14. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND OUTLIERS.

14.1 Precision -- The percent relative range (%RR) is used to assess the precision of the PAH measurements and is calculated using Equation 1.

Equation 1 % RR = 
$$\frac{2 * (X_1 - X_2)}{(X_1 + X_2)} * 100$$

Where:  $\begin{bmatrix} X_1 - X_2 \end{bmatrix}$  is the absolute value of the difference between the duplicate results

The overall precision of the data set at the 95% confidence level is calculated from the average of all the %RR values using Equation 2.

Equation 2 
$$\overline{P}_{95} = 2.51 * \underbrace{\sum_{i=1}^{n} %RR_i}_{n}$$

Where: %RR; is each individual percent relative range n = the number of duplicates

 $\overline{P}_{95}$  = 95% confidence level of the average precision

14.2 Accuracy -- The accuracy of the data set is determined from the analysis of the spiked samples. The accuracy for each PAH compound is calculated using Equation 3.

Equation 3 
$$A = 100 (Z - \overline{X})$$

Where: Z - is the analytical result in ng/L for the spiked sample

 $\overline{\mathbf{X}}$  - is the mean background concentration from the duplicate results

T - is the true value of the added spike

A - is the recovery for the added spike

The overall accuracy for each compound is the arithmetic mean over all the spiked samples, Equation 4.

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Equation 4 
$$\overline{A}j = \sum_{\substack{j=1 \\ n}}^{n} Aij$$

Where: Aij - is each recovery value for compound j

n - is the number of spiked samples

Aj - is the average recovery for compound j

The 95% confidence level for each mean recovery is computed using equation 5.

Equation 5 CL95 =  $\overline{A}_j \pm t(n-1, \alpha = 0.05) \cdot S$ 

Where: t(n-1, < = 0.05) is the appropriate two tailed students' t at < = 0.05

S - is the standard deviation associated with  $\overline{\mathsf{A}}_{j}$ 

CL95 - is the upper and lower 95% confidence limits of  $A_{\mbox{\scriptsize j}}$ 

- 14.3 Outliers -- An outlier is an extreme value, high or low, which has questionable validity as a member of the measurement set with which it is associated. Outliers may be rejected from the data set for the following reasons.
  - 14.3.1 A known experimental aberration occurred, such as instrument failure or there was an inconsistency in the procedure or technique.
  - 14.3.2 The t value for the datum is larger than the tabulated two tailed students' t for  $\alpha = 0.05$  at n-1 degrees of freedom. The t value is calculated using Equation 6.

Equation 6 
$$t = (\underline{Xi - \overline{X}})$$

Where:  $X_1$  - is the extreme value being tested

 $\overline{X}$  - is the mean of the measurement set for n observations

S - is the standard deviation associated with  $\overline{X}$ 

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If a value is rejected, the mean  $(\overline{X})$  and standard deviation are recalculated using the remaining data. This procedure can be reiterated using the next extreme value until no outliers remain.

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### 15. CORRECTIVE ACTION

15.1 Corrective action is initiated whenever the system is out of control. The following criteria are used to indicate out of control situations.

- 15.1.1 The area of a PAH internal standard is < 20,000 counts.
- 15.1.2 The recovery of a surrogate standard falls outside the range of  $R\pm 3s$  when R is the mean recovery and s is its associated standard deviation. This range is from 70% to 118% for 1-fluorobiphenyl at the beginning of this study and should be updated on a weekly basis.
- 15.1.3 The percent relative range for a given analyte of a duplicate pair exceeds 40% and the range is larger than the MDL for that analyte. This control limit is calculated using Equation 2 but substituting 3.27 for the constant 2.51 and should be updated after every fifth duplicate pair is analyzed.
- 15.1.4 The recovery for a spiked sample falls outside the range of  $A_j \pm t(n-1, \alpha=.01)$ \*S where  $t(n-1, \alpha=0.01)$  is the 99% two tailed t value for n-1 degrees of freedom. This range is from 48% to 118% at for all compounds the beginning of the study and should be updated for each compound after every fifth spike sample is analyzed.
- 15.2 If the out of control situation is due to an instrumental problem, the sample is reanalyzed after corrective action is completed. Results from the out of control analysis are discarded if the new analysis gives values that are in control.
- 15.3 If the out of control situation is due to other than instrumental problems, all samples analyzed between the last in control and present out of control sample are declared suspect and should be reanalyzed to ensure the validity of the data. This is just the out of control sample for the criteria in sections 15.1.1 and 15.1.2, and all samples run since the last in control duplicate for the criterion section 15.1.3, and all samples run since the last in control spike sample for the criterion in 15.1.4.
- 15.4 A log will be kept describing the out of control situations and the corrective action taken to remedy the situation.

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# 16. QUALITY ASSURANCE REPORTS TO MANAGEMENT

- 16.1 The analyst will identify and report any significant QA problems and recommend remedial steps to correct the problems.
- 16.2 At the end of the study, a report will be made that identifies the frequency of out of control situations and the necessary corrective action, the overall precision and accuracy of the data set, and the individual outliers.

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TABLE 1 ANTICIPATED SAMPLING SCHEDULE

Sample Set	Source	No. of Samples	Field Blanks	Duplicate	Triplicate/Spike <sup>a</sup>
1	Wells (12) and existing treatment (6)	18	1	1	1
2	Wells (12) and bench test (6)	18	1	1	1
3	Bench test	18	1	1	1
4	Bench test	18	1	1	1
5	Bench test	16	ľ	1	1
6	Bench test	16	1	1	1
7	Bench test	16	1	1	1
8	Wells (3) and pilot test (4)	7	1	1	1
9	Pilot test	4	1	-	-
10	Pilot test	4	1	1	1
11	Pilot test	4-	ľ		-
12	Pilot test	4	1	1	1
13	Pilot test	4	1	-	-
14	Pilot test	4	1	1	1
15	Pilot test	4	ľ	-	-
16	Pilot test	4	1	1	1
17	Pilot test	4	1	-	-
	TOTAL	163	1'7	12	12
	GRAND TOTAL	204 a	nalyses		

a One of the triplicates is spiked at the lab to give the spiked sample.

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TABLE 2 TARGET COMPOUNDS FOR GC/MS ANALYSES

		T	ONS	
Compound	CAS	Primary	Secondary	Source*
PNAs				
Acenaphthene	83-32-9	154	153,152	E,N,R
Acenaphthylene •	208-96-8	152	151,153	E,N,R
Anthracene	120-12-7	178	179,176	E,N,R
Benzo(a)anthracene	56-55-3	228	229,226	E,N,R
Benzo(b)fluoranthene	205-99-2	252	253,125	E,N.,R
Benzo(k)fluoranthene	207-08-9	252	253,125	E,N,R
Benzo(g,h,i <u>)</u> perylene	191-24-2	276	138,277	E,N
Benzo(a)pyrene	50-32-8	252	250,125	E,N,R
Benzo(e)pyrene	192-97-2	252	250,125	A,S
Chrysene	218-01-9	228	226,229	E,N,R
Dibenzo(a,h)anthracene	53-70-3	278	139,279	E,N
Fluoranthene	206-44-0	202	101,100	E,N,R
Fluorene	86-73-7	166	165,167	N₁,R
Indeno(1,2,3-cd)pyrene	193-39-5	276	138,277	N
1-Methylnaphthalene	90-12-0	142	141,1:1:5	A
2-Methy linaphtha lene	91-57-6	142	141,115	A
Naph tha lene	91-20-3	128	129,127	E,N₁,R
Perylene Perylene	198-55-0	252	250,126	A,S
Phenanthrene	85-01-8	178	179,176	E,N,R
Pyrene	129-00-0	202	101,100	E,N',R'
Triphenylene	217-59-4	228	226,229	A.
NITROGEN HETEROCYCLES				
Acridine	260-94-6	179	178,89	A,S
Carbazole	86-74-8	167	166,139	A,S
Indo le	120-72-9	117	90,89	A,S
Phenanthridine	229-87-8	179	178, 151	A
Quinoline	91-22-5	129	102,1:28	Α

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Table 2. Continued

			DNS	
Compound	<u>CAS</u>	Primary	Secondary	Source*
SULFUR HETEROCYCLES				
Benzo(b)thiophene	95-15-8	134	135,89	A
MI SCELLANEOUS				
Biphenyl 2,3-Dihydroindene Indene	92-52-4 496-11-7 95-13-6	154 118 116	153,76 179,91 175,89	A A A
AROMATIC AMINES**	•			-

<sup>\*</sup> E - EPA QC Check Samples N - NBS SRM-1647

R - EPA Repository Radian
A - Aldrich Chemical, Milwaukee, WI.
S - Sigma Chemical, St. Louis, Mo.

<sup>\*\*</sup> Up to 3; to be chosen after first round of testing.

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TABLE 3 MDL DATA FROM VALIDATION STUDY

COMPOUND	SPIKE LEVEL ng/L <sup>a</sup>	MEAN	STD.DEV.	% RECOVERY	WDLp	F_RATIO	WDL POOLEDC	MDL.
Naphtha lene	25 10	20.1 9.3	3.71 1.40	80 93	11.7 6.4	7.02	8.8	8.8
Acenaphthylene	25 10	19.9 7.4	1.22 0.39	80 74	3.8 1.8	9.78	-	1.8
Acenaphthene	25 10	20.4 8.3	1.54 0.39	82 83	4.8 1.8	15.6	-	1.8
Fluorene	25 10	22.5 7.6	1.21 1.00	90 76	4.2 4.5	1.46	3.2	3.2
Phenanthrene	25 10	20.4 9.8	3.35 3.00	81 98	10.5 13.6	1.24	9.1	9.1
Anthracene	25 10	18.1 8.0	3.99 0.70	72 80	12.6 3.1	32.5	-	3.1
Fluoranthene	25 10	23.1 9.4	2.33 1.2	93 94	7.3 5.4	3.77	5.7	5.7

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TABLE 3. continued MDL DATA FROM VALIDATION STUDY

COMPOUND S	SPIKE LEVEL ng/L <sup>a</sup>	MEAN	STD.DEV.	% RECOVERY	WDLp	F RATIO	MDL POOLEDC	MDL
Pyrene	25 10	24.0 11.4	2.31 1.0	96 114	7.3 4.5	5.34	5.6	5.6
Benzo(a)anthracene	25 10	21.6 10.9	3.10 1.0	86 109	9.7 4.5	9.61	-	4.5
Chrysene	25 10	19.6 9.5	3.71 0.5	67 95	11.7 2.3	55.	-	2.3
Benzo(b)Fluoranthre	ene 25 10	22.0 10.9	2.81 0.2	88 109	8.8 0.9	197.	-	0.9
Benzo(a)Pyrene	25 10	17.8 8.3	5.48 0.96	71 83	17.2 4.4	32.6	-	4.4
Indeno(123,cd)Pyrer	ne 25 10	20.3 8.8	2.39 0.9	81 88	7.5 4.1	7.05	5.7	5.7
Dibenzo(ah)Anthrace	ene 25 10	19.7 9.2	2.78 1.2	79 92	8.7 5.4	5.37	6.7	6.7
Benżo(ġhi)Pērylene	25 10	19.9 8.9	2.66 0.95	80 89	8.4 4.3	7.84	6.3	<u>6.3</u>
							Aver	āgē 4.7

a) seven replicates at 25 ng/L, 4 replicates at 10 ng/L b) Std. Dev. \* 3.143 at 25 ng/L; Std. Dev \* 4.541 at 10 ng/L c) pool if F ratio less than 8.94

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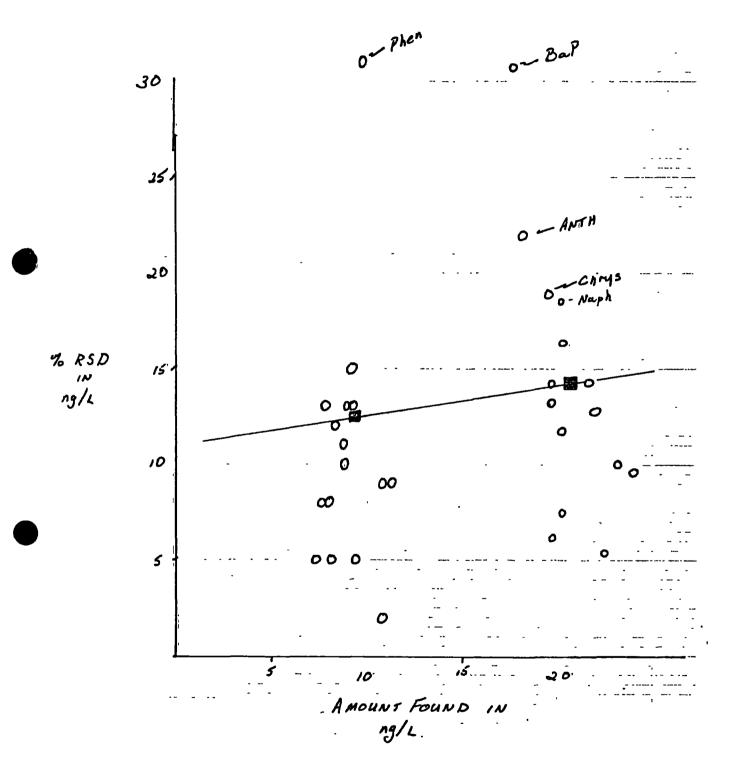


Figure 1. Relative Precision versus Concentration in the Validation Study.

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CH2M ## HILL	Montgomery, Office 807 South McDonough Street Montgomery, Alabama 36104
CLIENT	
SAMPLE NO.	
LOCATION	
DATE B	Υ

Figure 2. Sample Tag for Purgeables Sample.

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FIELD	TRACKING REPORT:	LOC-SN)	
FIELD SAMPLE CODE (FSC)	BRIEF DESCRIPTION	DATE TI	ME(s) SAMPLER
***************************************			
·		<u> </u>	
		-	
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Figure 3. Field Tracking Report Form.

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# **CHAIN OF CUSTODY RECORD**

	SAMPLERS (Signature)										
STATION NUMBER	STATION:LOCATION	DATE	TIME	DATE TIME		MPLE TYI	Pt Air	SEQ NO	NO OF		ANAL YSIS REQUIRED
				-  -							
			<u> </u>								
			-						-		
		<u> </u>									
							1				
			1					1			
Relinquisl	hed by: 15-granuel	<u>'</u>	Recei	ved by	/: (Signal	urel		,		Date/Time	
Relinquisl	hed by: (Signature)		Reline	quishe	d by.	Signaturi				Date/Time	
Relinquis	hed by: (Signature)		Received by sometimes							Date/Time	
Received by: Isoprature)			Received by Mobile Laboratory for field analysis: rs.ynaiure)						d	Date/Time	
Dispatched by: (Signature)			e/Time	Prime Received for Laboratory by:						Date/Time	
Method: d	f Shipment:	<del>!</del>	<u> </u>	<u>.                                    </u>							
		Distribution C	opy-Sur			eld Files					

Figure 4. Chain of Custody Report Form.